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☐ 1. Document ID: US 5833940 A

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US-PAT-NO: 5833940
DOCUMENT-IDENTIFIER: US 5833940 A

TITLE: Production of soluble silicates from biogenetic silica

DATE-ISSUED: November 10, 1998

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
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Mallow; William A.	Helotes	TX	N/A	N/A
Conner; Jesse R.	Darien	IL	N/A	N/A

US-CL-CURRENT: 423/334; 106/600, 106/601, 423/264, 423/332, 516/13

Full | Title | Citation | Front | Review | Classification | Date | Reference | Claims | KWIC | Draw Desc | Image

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L8: Entry 1 of 1 File: USPT Nov 10, 1998

DOCUMENT-IDENTIFIER: US 5833940 A
TITLE: Production of soluble silicates from biogenetic silica

ABPL:
Disclosed is a commercial grade of soluble silicate solutions, clear homogenous and water white essentially free of unreacted silica, made by dissolving in a closed container biogenetic silica, preferably rice hull ash, in a strong alkali solution, preferably sodium hydroxide in the presence of an agent, such as an active carbonaceous material, which prevents discoloration of the soluble silicates by absorbing and/or reacting with polyvalent metals, organic materials, and the like, in the biogenetic silica as it dissolves in and reacts with the alkali solution. The invention takes advantage of the residue of such active carbonaceous material on the biogenetic silica, such as rice hulls, left by commercial energy burning thereof which effectively prevents discoloration. A solid residue results from the method which is an active carbonaceous material including concentrated manganese from the biogenetic silica, both of which are valuable commercial products.

BSPR:
U.S. Pat. No. 1,293,008 (Blardone) discloses a boiling procedure for various lengths of time for producing a form of water glass from sodium silicate and sodium hydroxide; however, the boiling process cannot produce ratios higher than 1.5:1 of silica to sodium oxide. In subsequent paragraphs a fusion process is disclosed wherein sodium carbonate or sodium sulfate is fused with rice hull ash. While any ratio of sodium silicate desired can be produced by this fusion process, the energies adequate to couple sodium and silica at various ratios are at temperatures well over 2,000.degree. to 3,000.degree. F. These fusion products are then boiled in water to produce solutions, the open hearth process.

BSPR:
In obtaining soluble silicates from biogenetic silica, such as rice hull ash, in which the hull fibers have been burned off, the resulting soluble silicates have an amber color which is very difficult to remove. For example, attempts to remove the amber color proved inadequate by the following material and methods: activated carbon (perculation and filtration); activated, amorphous silica; zeolites (perculation and filtration); ion exchange resins; EDTA (ethylenediaminetetraacetic acid disodium salt); black rice hull ash (original and residual); PHPAA (partially hydrolyzed poly acrylic acid); sodium peroxide; chlorine; silica foam; silicate foam; and sodium gluconate.

BSPR:
Commercially available rice hull ash is prepared by burning rice hulls as an energy source in a furnace. In the process, raw rice hulls are continuously added to the top of the furnace and the ash is continuously removed from the bottom. Temperatures in the furnace range from 800.degree. to about 1400.degree. C., and the time factor for the ash in the furnace is about three minutes. Upon leaving the furnace, the ash is rapidly cooled to provide ease in handling. When treated by this method, silica remains in a relatively pure amorphous state rather than in the crystalline forms known as quartz, tridymite or crystobalite. Transition from the amorphous to the crystalline state generally takes place when the silica is held at very high temperatures, for example 2000.degree. C., or longer periods of time. The significance of having the silica in an amorphous state is that the silica ash maintains a porous skeletal structure rather than migrating to form crystals, and the amorphous form of silica does not cause silicosis thus reducing cautionary handling procedures. The burning of the rice hulls is time-temperature

related, and burning of these hulls under other conditions can be done so long as most of the ash is in an amorphous state with a porous skeletal structure.

BSPR:
On a commercial burning of rice hulls as an energy source, the resultant ash had the following chemical analysis (by weight):

BSPR:
The present invention comprises a hydration method of making soluble silicates such as sodium silicates by dissolving biogenetic silica in aqueous alkali solution such as sodium and potassium hydroxide in a closed container. By controlled burning of the rice hull ash, a "black ash" can be obtained with a residual carbonaceous content. This provides a method and material which, surprisingly, generates a clear, homogenous water white solution of alkali silicate when digested in aqueous sodium or potassium hydroxide in a closed container at temperatures and pressures which do not cause discoloration by the inherent organic material and trace minerals of the ash. Temperatures from ambient to the order of 275.degree. F. are suitable for most black ash. Higher temperatures and pressures may cause discoloration, for example, by the breakdown of the carbonaceous residue. While the mechanism of the prevention of the color formation is not known, it is possible that the carbonaceous residue in the ash is similar to "activated carbon" which may absorb or react with color forming agents before they are released to the alkali solution during the digestion of the ash. Surprisingly, perculation or filtration of amber colored sodium silicate through a bed or column of "black ash" did not remove the color. The isolated black residue recovered from the digestion of black ash in alkaline solution was also ineffective in removing color from an amber solution. Such amber solutions result from biogenetic silicas which contain less than 1% carbonaceous matter.

BSPR:
It is a further object of the present invention to provide a hydration method of producing a soluble silicate solution from rice hull ash having an active carbonaceous material dispersed throughout in an amount effective to prevent the discoloration of the soluble silicate during dissolution of the rice hull ash.

BSPR:
It is a further object of the present invention to provide a clear, homogenous soluble silicate solution free of unreacted silica made from dissolving in a closed container a biogenetic silica, such as rice hull ash, in a strong alkali solution effective to dissolve the biogenetic silica and produce the water soluble silicate in the presence of a carbonaceous material effective to prevent discoloration of the soluble silicate by extraneous matter in the rice hull ash, such as organic material, metal salts, and the like.

BSPR:
The biogenetic silica is obtained by the controlled burning of biogenetic materials containing silica, such as rice hulls, rice stalks, esquitum (horsetail weed), bagasse, certain bamboo palm leaves, particularly palmyra, pollen and the like. The burning of the biogenetic material is done under controlled conditions so that substantially all of the silica is in an amorphous rather than a crystalline state although minor amounts of crystalline silica can be present, as previously set forth. Preferably, the biogenetic materials are burned so that there is a residue of from about 2% to 8% of carbonaceous material present. In most commercial burnings, there will be approximately 0.5% to 8% or more of carbonaceous material (by weight) dispersed throughout the rice hull ash depending on the time and temperature of burning. It is only necessary to have sufficient carbon present to prevent discoloration. The rice hulls may be burned along with other biogenetic materials, such as wood chips, corn cobs and the like which increase the carbon residue. Excess carbon is not harmful to the reaction.

BSPR:
Advantageously, the biogenetic silica, such as rice hull ash, is dissolved in a closed container in a strong alkali solution effective to provide a solution of soluble silica, such as sodium or potassium silicate, at or above ambient temperature or atmospheric pressures or both. At elevated temperature and pressure, the reaction takes less time. Advantageously, the present invention does not require the use of high temperatures and pressures such as dissolving special grade quartz in a strong alkali solution as in the prior art processes. The strong alkali solution should have a pH of about 12 or greater. The alkali

can be pure sodium hydroxide or reaction products of calcium oxide and sodium carbonate or sodium hydroxides as by-product liquors and the like.

BSPR:
A series of experiments were performed on the dissolution of rice hull ash (RHA) in sodium hydroxide to form a solution of sodium silicate. There is no question that the RHA is being dissolved to a large degree by the sodium hydroxide and converted to a sodium silicate. While the products of these tests were not analyzed for silica, they were titrated for total alkali and total solids from which the silica was computed. In addition, the solutions were tested for gelling ability with dilute acid. All exhibited strong gelling, indicating the presence of substantial dissolved and/or colloidal silica/silicate. This dissolution of RHA occurs fairly rapidly and at low temperature and ambient pressure. In the absence of a discoloration preventive agent, the soluble silicate had an amber color which is undesirable for many commercial applications. This color appears to be due mostly to the presence of partially burned hulls or other organic matter, and/or small concentrations of metals, such as iron, manganese, copper or chromium intrinsic to the RHA. The discoloration was prevented by dissolving the RHA in the presence of a discoloration preventing agent, such as activated carbon. Advantageously, commercial energy burning of rice hulls leaves about a 21/2 to 8% by weight of a carbonaceous residue on the ash which absorbs or reacts with the organic matter and metals and thereby prevents this discoloration.

BSPR:
The following sets forth a series of experiments of the dissolution of rice hull ash (RHA) in substantially amorphous state in sodium hydroxide to form a solution of sodium silicate in the presence of about 2 1/2% to 8% (by weight) carbon.

DEPR:
The other biogenetic silica particles mentioned before can be substituted for rice hull silica in the above examples with substantially the same results.

CLPR:
9. A method of producing a clear soluble silicate solution free of unreacted silica from rice hull ash containing polyvalent metals and organic material which could leach into and contaminate and color the soluble silicate solution comprising,

CLPR:
10. The method of claim 9 where, the dissolution of the rice hull ash is at ambient temperature and atmospheric pressure.

CLPR:
11. The method of claim 9 where, the dissolution of the rice hull ash is at a temperature above ambient temperature.

CLPR:
13. The method of claim 9 where, the dissolution of the rice hull ash is at a temperature above ambient temperature and at a pressure above atmospheric pressure.

CLPV:
dissolving in a closed container the rice hull ash in an alkali solution of at least pH 12 in a molar ratio of silica to alkali in the range of 1 to 2 to 2 to 1 and at a temperature not higher than 275.degree. F. in the presence of a solid carbonaceous material thereby preventing the polyvalent metals and organic material from leaching into and coloring the resulting soluble silicate solution,